

Metallography and Phase Relations – Study of microstructures that may be observed in metals and alloys using visible light microscopy, scanning electron microscopy, and other techniques, how those microstructures are produced, as well as the relationships between structural properties and failures. Required for the BS degree in MSNE.

- **Instructor:** Peter Loos **on campus:** ME 215 **email:** ploos@rice.edu
- **Office hours:** After class, by appointment, and others TBD
- **Class Meets:** TuTh 1:00-2:15 pm in HBH 453. Some additional meeting time will be scheduled for operation of the instruments, hopefully Tu and/or Th either just before or just after the scheduled class period.
- **Prerequisites:** MSNE 301 is required. Completion of MSNE 303 is also assumed.
- **Text:** None. There will be several reading assignments from the published literature.
- **Grading:** 35% homework, 25% tests, 15% quizzes, 20% presentations, 5% vocabulary list. There will be approximately two tests, three quizzes, seven homework assignments, and one presentation. Points may be deducted for assignments turned in late and for bad or missing references.
- **References:** When using information from a source other than class lecture slides or your own original work, a proper reference must be given. Use ACS format per pubs.acs.org/doi/pdf/10.1021/bk-2006-STYG.ch014 or in simpler form: <http://library.williams.edu/citing/styles/acs.php>.
- **Honor Code:** For every assignment students must do all the writing, typing, calculating, and analyzing themselves, must **not** look at or copy from any work done by any student in this class (past or present), and must include and sign the Rice Honor Pledge on work turned in. [Plagiarism](#) and other academic fraud will be addressed per the [Honor System Handbook](#). For homework, presentations, and vocabulary lists, students are encouraged to communicate with each other, the Instructor, or anyone else with knowledge of the subject. But for tests and quizzes no communication is allowed except with Instructor.
- **Absence Policy:** Missed quizzes and presentations must be made up right away. Students are expected to attend class. The slides posted on Canvas do not include everything said in class.
- **Software:** Students will use spreadsheets such as MS Excel or [OpenOffice Calc](#) for some of the calculations on homework and tests. Also, MS PowerPoint or OpenOffice [Impress](#) or similar software can be used for presentations. For compatibility, all files should be saved in either XLS, DOC, PPT, or PDF format.
- **Disability Support:** Any student with a disability requiring accommodations in this course is encouraged to contact Instructor after class or by email. Additionally, students need to contact [Disability Support Services](#) in Allen Center.

Lecture Topics

- History of microscopy, metallurgy, and metallography including [H.C. Sorby](#)'s sample preparation and microscopy in 1863 and [H.W.B. Rozeboom](#)'s Fe-Fe₃C phase diagram in 1900.
- Alloys of interest: iron, steel, stainless steel, aluminum, titanium, etc.
- Length scales of interest
- Purposes of metallography: R&D, quality, reliability, yield, process development, process control, etc.
- Sample preparation techniques: sectioning, mounting, grinding, polishing, and etching (as needed) to reveal the true structure without changing it. Etchants for specific applications
- Safety in the metallurgy lab: machinery as well as corrosive and poisonous chemicals
- Macrostructure / macroetch / macroanalysis of cast and wrought materials. Dye penetrant and magnetic particle inspection, sulfur print and related methods, hydrogen evolution, blue etch anodizing of Ti alloys.
- Visible light microscopy: optical components, magnification, resolution, enhancing resolution through colored filters and oil immersion, bright field/dark field, polarizing filters, interference contrast.
- Scanning electron microscopes: electron guns; thermionic and field emission; electron wavelength; resolution, magnetic lenses; condenser system; raster scan; optimizing spot size. Beam alignment and astigmatism.
- Beam-specimen interaction, scattering of electrons by atoms – six scattering mechanisms, electron trajectories, ranges for electron penetration and x-ray generation.
- Generation & properties of: backscattered, secondary, & Auger electrons; characteristic & Bremsstrahlung x-rays; cathodoluminescence, voltage contrast, magnetic contrast, thermal wave (scanning electron acoustic) microscopy
- X-ray microanalysis: quantitative analysis, ZAF corrections, minimum detectable amount of an element, energy dispersive vs. wavelength dispersive x-ray detectors, optimizing characteristic x-ray production
- SEM sample preparation for various purposes, surface replicas.
- Kikuchi lines and electron backscatter diffraction (EBSD)
- Quantitative metallography: ASTM E112 grain size, lognormal grain size distribution, defects, etc.
- Hot work, cold work, and recrystallization. Electron channeling contrast.
- Binary phase diagrams: eutectic, eutectoid, peritectic, peritectoid, monotectic, etc.; cooling paths, precipitates formed. Miscibility gap, congruent melting. Coring. Dendritic structures. Pearlite and lamellar structures.

- Isothermal transformation and continuous cooling transformation diagrams for various alloys, and their use.
- The five strengthening mechanisms. Carburizing, nitriding, decarburization.
- The hierarchy of authoritative documents. These include plenty of optical and other forms of metallography but not SEM, TEM, EDX, or WDX.
- Field failures: sample preservation, types of failures, and how to recognize them.
- Identifying engineering problems.

Tentative list of possible hands-on lab-type activities:

- (1) Section, mount, grind, polish, etch, and photograph a Ni alloy specimen on the visible light microscope.
- (2) Grain size analysis of single-phase and/or two-phase materials.
- (3) Sample cleaning, sputter coating, and surface replication, to dissipate charge and improve resolution.
- (4) Calibration of SEM magnification and EDX energy using standard samples.
- (5) Use of detectors for secondary electrons and backscattered electrons. Contrast due to topography, composition, and electron channeling.
- (6) EDX point analysis, line scans, and area maps. Use of ZAF correction with EDX data
- (7) Voltage contrast microscopy of an electronic circuit switching between two states.
- (8) Electron backscatter diffraction for polycrystalline specimens.
- (9) Wavelength dispersive x-ray analysis using crystal spectrometers.
- (10) Texture analysis (preferred orientation) by x-ray diffraction, displayed as pole figures.

(1-2) uses various equipment in Ryon B08. (3) uses [Emitech K550](#) sputter coater on GRB 2nd floor. (4)-(7) use JEOL 6460 and [NORAN System 6](#) in GRB 227 or [FEI Quanta 400](#) and EDAX in Butcher 160A. (8) uses [FEI Helios](#) and EBSD in Brockman B05. (9) uses [JEOL JXA-8530F](#) in KWG 329-330. (10) uses Rigaku [SmartLab](#) in Butcher 160.

Goals for this course:

To address the required ABET Student Outcomes, students will have an ability to:

1. identify, formulate, and solve complex engineering problems by applying principles of engineering, science, and mathematics
3. an ability to communicate effectively with a range of audiences
6. an ability to develop and conduct appropriate experimentation, analyze and interpret data, and use engineering judgment to draw conclusions

and to address one of the ABET Program Criteria, this course prepares graduates to:

- integrate the understanding of the scientific and engineering principles underlying the four major elements of the field: structure, properties, processing, and performance ...

More specifically, students will become familiar with

- a) sample preparation methods and equipment for microstructure and macrostructure evaluation
- b) microscopy methods and equipment used to observe those microstructures, particularly the visible light microscope (VLM) and scanning electron microscope (SEM) .
- c) many of the microstructures which occur in metals and alloys, how those result from composition and thermal history of the material,
- d) use of equilibrium phase diagrams and nonequilibrium transformation diagrams to predict and understand microstructure and thermal history
- e) the terminology used to describe microstructures
- f) how problems in the microstructure or macrostructure can lead to failure

References

ASM Handbook Volume 3, [Alloy Phase Diagrams](#) (1992), **Volume 9, [Metallography and Microstructures](#)** (2004), **Volume 11, [Failure Analysis and Prevention](#)** (2002), **Volume 12, [Fractography](#)** (1987)

Metallographic Etching by G. Petzow, ASM (1978)

Metallography of Phase Transformations by G. A. Chadwick, Butterworths (1972) which is out of print

Annual Book of ASTM Standards includes about 444 standards for metallography. See [search results](#). Fondren Library has these on microfilm. Also ASTM has published many hundreds of Special Technical Publications (STP) about metallography. See [search results](#). Fondren has many of these on the shelves.

SAE standards concerning metallography, see [search results](#) after clicking on "Standard".

Scanning Electron Microscopy and X-Ray Microanalysis: A Text for Biologists, Materials Scientists, and Geologists 2nd Ed. by Goldstein, Newbury, Echlin, Joy, Fiori, & Eric Lifshin (1981)

Electron Microscopy and Analysis 3rd Ed. by Goodhew, Humphreys, & Beanland (2001)

Resources On the Web

- Interactive binary phase diagrams with accompanying photomicrographs from Virginia Tech and Cambridge Univ.:

www.sv.vt.edu/classes/MSE2094_NoteBook/96ClassProj/examples/kimcon.html (use the [Wayback Machine](#))
www.doitpoms.ac.uk/miclib/phase_diagrams.php

- Microscopy U from Nikon www.microscopyu.com/index.html

Journals:

www.sciencedirect.com.ezproxy.rice.edu/science/journal/00260800 Metallography
www.sciencedirect.com.ezproxy.rice.edu/science/journal/10445803 Materials Characterization
link.springer.com.ezproxy.rice.edu/journal/volumesAndIssues/11508 The Physics of Metals and Metallography

Videos:

www.youtube.com/user/MaterialsScience2000 Several good videos listed
www.youtube.com/watch?v=fc8zrgYJCJw Metallography Part I - Macroscopic Techniques
www.youtube.com/watch?v=UuHofNW40Yw Metallography Part II - Microscopic Techniques
www.youtube.com/watch?v=GY9lfO-tVfE SEM
www.youtube.com/watch?v=7Z90OZ7C2jI Vickers Hardness Test
www.youtube.com/watch?v=xEK-c1pkTUI Dye Penetrant Inspection

Metallography:

www.msm.cam.ac.uk/phase-trans/abstracts/CP1b.html Sample preparation for metallography.
www.doitpoms.ac.uk/tlplib/microstructural_exam/ Microstructural Examination
www.doitpoms.ac.uk/tlplib/optical-microscopy/index.php Optical Microscopy and Specimen Preparation
ASTM E7-03 Standard Terminology Relating to Metallography
ASTM Committee E04 on Metallography Committee E04 on Metallography
www.astm.org/DIGITAL_LIBRARY/STP/PAGES/STP25092S.htm History of Committee E4 on Metallography

Metallurgy:

www.doitpoms.ac.uk/tlplib/casting/index.php Casting
www.doitpoms.ac.uk/tlplib/crystallographic_texture/index.php Crystallographic Texture
www.doitpoms.ac.uk/tlplib/artefact/index.php Examination of a Manufactured Article
www.doitpoms.ac.uk/tlplib/phase-diagrams/index.php Phase diagrams and solidification
www.doitpoms.ac.uk/tlplib/solid-solutions/index.php Solid Solutions
www.doitpoms.ac.uk/tlplib/solidification_alloys/index.php Solidification of Alloys